

(19)

Europäisches Patentamt
European Patent Office
Office européen des brevets



(11)

EP 1 500 799 A1

(12)

EUROPEAN PATENT APPLICATION
published in accordance with Art. 158(3) EPC

(43) Date of publication:
26.01.2005 Bulletin 2005/04

(51) Int Cl.7: **F01N 3/02**

(21) Application number: **03747537.3**

(86) International application number:
PCT/JP2003/004480

(22) Date of filing: **09.04.2003**

(87) International publication number:
WO 2003/093658 (13.11.2003 Gazette 2003/46)

(84) Designated Contracting States:
**AT BE BG CH CY CZ DE DK EE ES FI FR GB GR
HU IE IT LI LU MC NL PT RO SE SI SK TR**

(72) Inventor: **YAMADA, Keiji**
c/o **IBIDEN CO., LTD.** Ogakikita Kojo
Ibi-gun, Gifu 501-0695 (JP)

(30) Priority: **11.04.2002 JP 2002109717**

(74) Representative: **Hart Davis, Jason et al**
Cabinet Beau de Loménie,
158, rue de l'Université
75340 Paris Cedex 07 (FR)

(71) Applicant: **IBIDEN CO., LTD.**
Ogaki-shi Gifu-ken 503 (JP)

(54) HONEYCOMB FILTER FOR CLARIFYING EXHAUST GAS

(57) An object of the present invention is to provide a honeycomb filter for purifying exhaust gases that is free from a gap formed between a plug and a partition wall and cracks generated in the plug and a portion of the partition wall contacting the plug, and is superior in durability.

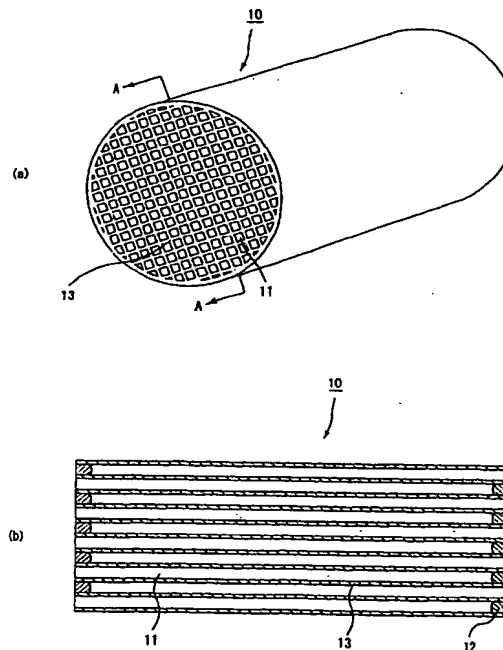
The present invention provides a honeycomb filter for purifying exhaust gases which has a structure in which:

a columnar body made of porous ceramic comprises a number of through holes, said through holes being placed in parallel with one another in the length direction with a wall portion interposed therebetween;

predetermined through holes of said through holes are filled with plugs at one end of said columnar body, while the through holes that have not been filled with said plugs at said one end are filled with plugs at the other end of said columnar body; and a part or all of said wall portion functions as a filter for collecting particulates,

wherein the porosity of the columnar body is in a range from 20 to 80%, and the porosity of the plug is 90% or less and is also set to 0.15 to 4.0 times as much as the porosity of the columnar body.

Fig. 1



EP 1 500 799 A1

support an oxidizing catalyst in its pores, hydrocarbon contained in exhaust gases that flow into the honeycomb filter is made to react with the oxidizing catalyst so that heat generated through this reaction is utilized for the regenerating process of the honeycomb filter. In the honeycomb filter that carries out the regenerating process in this manner, it is necessary to increase the porosity thereof, because clogging of pores tends to occur due to particulates since the oxidizing catalyst is supported on the inside of each pore of the honeycomb filter, and because the oxidizing catalyst needs to be supported as much as possible in order to generate a large amount of heat.

[0014] In such a honeycomb filter with high porosity, however, the difference between the coefficient of thermal expansion of the columnar body and the coefficient of thermal expansion of the plug having a compact structure becomes greater, with the result that a gap tends to occur between the plug and the partition wall and cracks tend to occur in the plug and the portion of the partition wall contacting the plug due to thermal stresses occurring between the plug and the partition wall, which are caused by the firing process upon manufacturing and high-temperature exhaust gases during operation, as described above.

[0015] In addition, in order to allow such a honeycomb filter with high porosity to support a catalyst, normally, a method is used in which: the honeycomb filter is coated with γ -alumina or the like having a high specific surface area to form a catalyst supporting film and noble metal which works as the catalyst is dispersed and supported on the catalyst supporting film; however, in this method, the catalyst supporting film tends to be formed so as to intrude between the plug and the portion of the partition wall contacting the plug, and cracks or the like tend to occur due to thermal stresses caused by the difference between the coefficients of expansion of these materials.

[0016] Conventionally, a honeycomb filter in which a plug is allowed to have gas permeability so as to easily separate particulates collected from exhaust gases upon back-washing; and a honeycomb filter in which the porosity of a plug is limited so as to improve the purifying performance (see JP Kokai 2003-3823): have been proposed. However, these honeycomb filters have not been prepared by taking the above-mentioned problems into consideration, and the objects of these are completely different from that of the present invention; therefore, the technical premises thereof are completely different from that of the present invention.

SUMMARY OF THE INVENTION

[0017] The present invention is made to solve the above-mentioned problems, and its object is to provide a honeycomb filter for purifying exhaust gases that is free from a gap formed between a plug and a partition wall and cracks generated in the plug and a portion of the partition wall contacting the plug, and is superior in durability.

[0018] The present invention provides a honeycomb filter for purifying exhaust gases which has a structure in which:

a columnar body made of porous ceramic comprises a number of through holes, said through holes being placed in parallel with one another in the length direction with a wall portion interposed therebetween;

predetermined through holes of said through holes are filled with plugs at one end of said columnar body, while the through holes that have not been filled with said plugs at said one end are filled with plugs at the other end of said columnar body; and

a part or all of said wall portion functions as a filter for collecting particulates

wherein the porosity of the columnar body is in a range from 20 to 80%, and the porosity of the plug is 90% or less and is also set to 0.15 to 4.0 times as much as the porosity of the columnar body.

[0019] Moreover, in the case where a catalyst is supported on the honeycomb filter for purifying exhaust gases of the present invention, the porosity of the columnar body and the plug is desirably measured after a catalyst supporting film has been formed on the honeycomb filter for purifying exhaust gases of the present invention.

BRIEF DESCRIPTION OF THE DRAWINGS

[0020]

Fig. 1(a) is a perspective view that schematically shows one example of a honeycomb filter for purifying exhaust gases of the present invention, and Fig. 1(b) is a longitudinal cross-sectional view taken along line A-A of Fig. 1(a). Fig. 2 is a perspective view that schematically shows another example of the honeycomb filter for purifying exhaust gases of the present invention.

Fig. 3(a) is a perspective view that schematically shows a porous ceramic member to be used for the honeycomb filter for purifying exhaust gases of the present invention shown in Fig. 2, and Fig. 3(b) is a longitudinal cross-sectional view taken along line B-B of Fig. 3(a).

Fig. 4(a) is a cross-sectional view that schematically shows a mouth sealing treatment to be carried out upon manufacturing the honeycomb filter for purifying exhaust gases of the present invention, and Fig. 4(b) is a partial

with partition wall 13 being interposed therebetween, and the porosity of the columnar porous ceramic members is desirably set to 20 to 80%. When the porosity of the columnar body is less than 20%, the honeycomb filter 10 is more likely to generate clogging, while the porosity of the columnar body exceeding 80% causes degradation in the strength of the honeycomb filter 10, with the result that it might be easily broken.

[0031] Here, the above-mentioned porosity can be measured through known methods such as a mercury press-in method, Archimedes method, a measuring method using a scanning electronic microscope (SEM), and the like.

[0032] With respect to the size of the columnar body, not particularly limited, it is appropriately determined by taking the size of an exhaust gas passage of the internal combustion engine and the like to be used into consideration. Moreover, with respect to the shape thereof, not particularly limited as long as it is a column shape, for example, any desired shape such as a cylinder shape, an elliptical column shape, a rectangular column shape and the like may be used, and in general, as shown in Fig. 1, those having a cylinder shape are often used.

[0033] With respect to the porous ceramics constituting the columnar body, not particularly limited, examples thereof include: oxide ceramics such as cordierite, alumina, silica, mullite and the like; carbide ceramics such as silicon carbide, zirconium carbide, titanium carbide, tantalum carbide, tungsten carbide and the like; and nitride ceramics such as aluminum nitride, silicon nitride, boron nitride, titanium nitride and the like. However, normally, oxide ceramics such as cordierite and the like are utilized. This is because these materials make it possible to carry out the manufacturing process at low costs, have a comparatively small coefficient of thermal expansion and are less likely to be oxidized during use. Further, silicon-containing ceramics made by blending metallic silicon in the above-mentioned ceramics, and ceramics bonded by silicon and silicate compound may also be used.

[0034] The average pore diameter of the columnar body is desirably set in a range from 5 to 100 μm . The average pore diameter of less than 5 μm tends to cause clogging of particulates easily. In contrast, the average pore diameter exceeding 100 μm tends to cause particulates to pass through the pores, with the result that the particulates cannot be collected, making the columnar body unable to function as a filter.

[0035] Here, the honeycomb filter 10 of the present invention has a structure in which a plug 12 is injected to each through hole 11 at one of the ends of the columnar body or the other end thereof, and the porosity of this plug 12 is set to 90% or less. The porosity of the plug 12 exceeding 90% causes degradation in the strength of the plug 12, with the result that the plug 12 might be easily broken due to high-temperature exhaust gases that flow into the honeycomb filter 10 and a thermal impact and the like caused by thermal cycles and the like during regenerating processes. Moreover, even when the plug 12 is not broken, particulates tend to pass through the plug 12, making the columnar body unable to sufficiently function as an exhaust gas purifying filter.

[0036] With respect to the multiplying ratio of the porosity of the plug 12 to the porosity of the columnar body, the lower limit thereof is set to 0.15 times, and the upper limit thereof is set to 4.0 times.

[0037] When the lower limit of the multiplying ratio of the porosity of the plug 12 to the porosity of the columnar body is less than 0.15 times, the difference between the coefficient of thermal expansion of the columnar body and the coefficient of thermal expansion of the plug 12 becomes too large, with the result that during a firing process upon manufacturing, a gap tends to occur between the plug 12 and the wall portion 13 and cracks tend to occur in the plug 12 and a portion of the wall portion 13 that contacts the plug 12 due to the difference in the coefficients of thermal expansion. In contrast, when the upper limit of the multiplying ratio of the porosity of the plug 12 to the porosity of the columnar body exceeds 4.0 times, the difference between the coefficient of thermal expansion of the columnar body and the coefficient of thermal expansion of the plug 12 becomes too large in this case also, with the result that during a firing process upon manufacturing, a gap tends to occur between the plug 12 and the wall portion 13 and cracks tend to occur in the plug 12 and a portion of the wall portion 13 that contacts the plug 12 due to the difference in the coefficients of thermal expansion; presumably causing a reduction in the strength.

[0038] Here, the lower limit of the multiplying ratio of the porosity of the plug 12 to the porosity of the columnar body is desirably set to 0.25 times, and the upper limit thereof is desirably set to 1.5 times.

[0039] In the case where the lower limit of the multiplying ratio of the porosity of the plug 12 to the porosity of the columnar body is less than 0.25 times, even if the difference between the coefficient of thermal expansion of the columnar body and the coefficient of thermal expansion of the plug 12 is comparatively large with neither the gap nor cracks occurring during a firing process upon manufacturing, a gap tends to occur between the plug 12 and the wall portion 13 and cracks tend to occur in the plug 12 and a portion of the wall portion 13 that contacts the plug 12 due to thermal cycles repeatedly applied thereto by high-temperature exhaust gases during operation and heating processes at the time of the regenerating process, causing a reduction in the strength. Moreover, in the case where a catalyst supporting film is formed on the honeycomb filter of the present invention, the catalyst supporting film is sometimes formed between the plug 12 and a portion of the wall portion 13 that contacts the plug 12 so as to intrude therebetween; consequently, in this case, cracks also occur due to a thermal stress caused by the difference between the coefficients of thermal expansion of the catalyst supporting film and the plug 12 as well as a portion of the wall portion 13 that contacts the plug 12, resulting in a reduction in strength.

[0040] In contrast, in the case where the upper limit of the multiplying ratio of the porosity of the plug 12 to the porosity

porous ceramic members 30 has a structure in which a number of through holes 31 are arranged in parallel with one another in the length direction so that the partition wall 33 that separates the through holes 31 from each other function as filters.

[0051] In other words, as shown in Fig. 3 (b), each of the through holes 31 formed in the porous ceramic member 30 has either of its ends on the inlet side or outlet side of exhaust gases sealed with a plug 32; thus, exhaust gases that have entered one of the through holes 31 are allowed to flow out of another through hole 31 after having always passed through the partition wall 33 that separates the corresponding through holes 31.

[0052] Moreover, the sealing material layer 26, which is formed on the periphery of the ceramic block 25, is placed so as to prevent exhaust gases from leaking through the peripheral portion of each ceramic block 25 when the honeycomb filter 20 is installed in an exhaust passage of an internal combustion engine.

[0053] Here, in Fig. 3 (b), arrows indicate flows of exhaust gases.

[0054] The honeycomb filter 20 having the above-mentioned structure is placed in an exhaust gas purifying device that is installed in the exhaust passage in an internal combustion engine so that particulates in the exhaust gases discharged from the internal combustion engine are captured by the partition wall 33 when passing through the honeycomb filter 20; thus, the exhaust gases are purified.

[0055] Since the honeycomb filter 20 of this type has superior heat resistance and enables easy regenerating processes and the like, it has been applied to various large-size vehicles and vehicles with diesel engines.

[0056] In the honeycomb filter 20 of the present invention having the above-mentioned structure, the porosity of the columnar body (the member corresponding to the ceramic block 25 from which the plug 32 is omitted) is set in a range from 20 to 80%, and the porosity of the plug 32 is 90% or less, and in this structure, with respect to the multiplying ratio of the porosity of the plug 32 to the porosity of the columnar body, the lower limit is 0.15 times, and the upper limit is 4.0 times. Thus, the structure is the same as the honeycomb filter 10 explained by reference to Fig. 1.

[0057] Moreover, in the same manner as the plug 12 explained in the honeycomb filter 10 of the present invention, with respect to the multiplying ratio of the porosity of the plug 32 to the porosity of the columnar body, the lower limit is desirably set to 0.25 times, and the upper limit is desirably set to 1.5 times, with the lower limit of the porosity of the plug 32 corresponding to 3%, and the plug 32 is desirably made of porous ceramics.

[0058] With respect to the material for the porous ceramic member 30, not particularly limited, for example, the same materials as the ceramic material constituting the columnar body of the honeycomb filter 10 of the present invention may be used. Among these, silicon carbide, which has great heat resistance, superior mechanical properties and great thermal conductivity, is desirably used.

[0059] With respect to the particle size of ceramic particles to be used upon manufacturing the porous ceramic members 30, although not particularly limited, those which are less likely to shrink in the succeeding firing process are desirably used, and for example, those particles, prepared by combining 100 parts by weight of particles having an average particle size from 0.3 to 50 μm with 5 to 65 parts by weight of particles having an average particle size from 0.1 to 1.0 μm , are desirably used. By mixing ceramic powders having the above-mentioned respective particle sizes at the above-mentioned blending ratio, it is possible to provide a porous ceramic member 30.

[0060] In the honeycomb filter 20 of the present invention, a plurality of porous ceramic members 30 of this type are combined with one another through sealing material layers 24 to constitute a ceramic block 25, and a sealing material layer 26 is also formed on the periphery of the ceramic block 25.

[0061] In other words, in the honeycomb filter 20 of the present invention, the sealing material layer is formed between the porous ceramic members 30 as well as on the periphery of the ceramic block 25, and the sealing material layer (sealing material layer 24) formed between the porous ceramic members 30 functions as an adhesive layer for bonding the porous ceramic members 30 to one another, while the sealing material layer (sealing material layer 26) formed on the periphery of the ceramic block 25 functions as a sealing member for preventing leak of exhaust gases from the passage of an internal combustion engine.

[0062] With respect to the material forming the sealing material layer (sealing material layer 24 and sealing material layer 26), not particularly limited, for example, a material composed of an inorganic binder, an organic binder, inorganic fibers and inorganic particles may be used.

[0063] As described above, in the honeycomb filter 20 of the present invention, the sealing material layer is formed between the porous ceramic members 30 as well as on the periphery of the ceramic block 25; and these sealing material layers (sealing material layer 24 and sealing material layer 26) may be made of the same material or different materials. In the case where the same material is used for the sealing material layers, the blending ratio of the material may be the same or different.

[0064] With respect to the inorganic binder, for example, silica sol, alumina sol and the like may be used. Each of these may be used alone or two or more kinds of these may be used in combination. Among the inorganic binders, silica sol is more desirably used.

[0065] With respect to the organic binder, examples thereof include polyvinyl alcohol, methyl cellulose, ethyl cellulose,

coefficient of thermal expansion of the plug is not so big; therefore, it becomes possible to prevent a gap from occurring between the plug and the partition wall and also to prevent cracks from occurring in the plug and the portion of the partition wall contacting the plug due to accumulated thermal stresses and the like caused by thermal cycles, thereby making the honeycomb filter of the present invention superior in the durability.

[0078] Next, the following description will be given of one example of a manufacturing method for the above-mentioned honeycomb filter of the present invention.

[0079] In the case where the honeycomb filter of the present invention has a structure, as shown in Fig. 1, in which the entire structure is constituted by a single sintered body, first, an extrusion-molding process is carried out by using the material paste mainly composed of the above-mentioned ceramics so that a ceramic formed body having almost the same shape as shown by a honeycomb filter 10 of Fig. 1 is manufactured.

[0080] With respect to the material paste, not particularly limited, any material paste may be used as long as the porosity of the columnar body after the manufacturing process is set in a range from 20 to 80%, and, for example, a material paste, prepared by adding a binder and a dispersant solution to powder made of the above-mentioned ceramics, may be used.

[0081] With respect to the above-mentioned binder, not particularly limited, examples thereof include methylcellulose, carboxy methylcellulose, hydroxy ethylcellulose, polyethylene glycol, phenol resins, epoxy resins and the like.

[0082] Normally, the blend ratio of the above-mentioned binder is desirably set to 1 to 10 parts by weight to 100 parts by weight of ceramic powder.

[0083] With respect to the above-mentioned dispersant solution, not particularly limited, for example, an organic solvent such as benzene or the like, alcohol such as methanol or the like, water and the like may be used.

[0084] An appropriate amount of the above-mentioned dispersant solution is blended so that the viscosity of the material paste is set in a predetermined range.

[0085] These ceramic powder, binder and dispersant solution are mixed by an attritor or the like, and sufficiently kneaded by a kneader or the like, and then extrusion-molded so that the above-mentioned ceramic formed body is formed.

[0086] Moreover, a molding auxiliary may be added to the above-mentioned material paste, based on necessity.

[0087] With respect to the molding auxiliary, not particularly limited, examples thereof include: ethylene glycol, dextrin, fatty acid soap, polyalcohol and the like.

[0088] Furthermore, a pore forming agent, such as balloons that are fine hollow spheres composed of oxide-based ceramics, spherical acrylic particles and graphite, may be added to the above-mentioned material paste, based on necessity.

[0089] With respect to the above-mentioned balloons, not particularly limited, for example, alumina balloons, glass micro-balloons, shirasuballoons, flyashballoons (FABalloons) and mullite balloons may be used. Among these, fly ash balloons are more desirably used.

[0090] Next, the above-mentioned ceramic formed body is dried by using a dryer, such as a microwave dryer, a hot-air dryer, a dielectric dryer, a decompression dryer, a vacuum dryer, a freeze dryer and the like, and predetermined through holes are then filled with a filler paste that is going to be a plug; thereafter, the above-mentioned through holes are subjected to mouth sealing processes so as to be sealed.

[0091] Fig. 4(a) is a cross-sectional view that schematically shows an example of a mouth sealing device to be used in the above-mentioned mouth sealing process, and Fig. 4 (b) is a partial enlarged cross-sectional view that shows one portion thereof.

[0092] As shown in Fig. 4, a mouth sealing device 100 to be used in the mouth sealing process has a structure in which: a pair of tightly-closed filler discharging tanks 110, each of which has a mask 111 that has an opening section 111a having a predetermined pattern and is placed on its side face, are filled with filler paste 120 and arranged so that the two side faces, each having the mask 111, are aligned face to face with each other.

[0093] In the case where the mouth sealing process of the ceramic dried body is carried out by using the mouth sealing device 100 of this type, first, a ceramic dried body 40 is secured between the filler discharging tanks 110 so that the end face 40a of the ceramic dried body 40 is made in contact with the mask 111 formed on the side face of each of the filler discharging tanks 110.

[0094] At this time, the opening section 111a of the mask 111 and the through hole 42 of the ceramic dried body 40 are positioned so that they are aligned face to face with each other.

[0095] Next, a predetermined pressure is applied to the filler discharging tank 110 by using, for example, a pump such as a mono-pump or the like, so that the filler paste 120 is discharged from the opening section 111a of the mask 111; thus, by injecting the filler paste 120 to the end of the through hole 42 of the ceramic dried body 40, predetermined through holes 42 of the ceramic dried body 40 are filled with the filler paste 120 that forms the plugs.

[0096] Here, the mouth sealing device to be used in the above-mentioned mouth sealing process is not limited to the above-mentioned mouth sealing device 100, for example, another system may be used in which: an open-type filler discharging tank in which a stirring member is installed is prepared, and by moving the stirring member up and

[0111] In the case where the honeycomb filter of the present invention has a structure in which a plurality of porous ceramic members are combined with one another through sealing material layers, as shown in Fig. 2, first, an extrusion-molding process is carried out by using a material paste mainly composed of ceramics as described earlier so that a raw molded body, which has a shape corresponding to the porous ceramic member 30 as shown in Fig. 3, is formed.

[0112] Here, with respect to the above-mentioned material paste, the same material paste as explained in the honeycomb filter constituted by a single sintered body may be used.

[0113] Next, the above-mentioned raw molded body is dried by using a microwave dryer or the like to become a dried body, and predetermined through holes are then filled with a filler paste that is going to be plugs; thereafter, the above-mentioned through holes are subjected to mouth sealing processes so as to be sealed.

[0114] Here, with respect to the filler paste, the same filler paste as explained in the honeycomb filter constituted by a single sintered body may be used, and with respect to the mouth sealing processes, the same method as the above-mentioned honeycomb filter 10 may be used, except that the subject to be filled with the filler paste is different.

[0115] Next, the dried body that has been subjected to the mouth seal ingproce ses is subjected to degreas ing and firingprocesses under predetermined conditions so that a porous ceramic member having a structure in which a plurality of through holes are placed in parallel with one another in the length direction with partition wall interposed therebetween is manufactured.

[0116] Here, with respect to the degreasing and sintering conditions and the like of the above-mentioned molded product, the same conditions as those conventionally used for manufacturing a honeycomb filter in which a plurality of porous ceramic members are combined with one another through sealing material layers may be applied.

[0117] Next, as shown in Fig. 5, porous ceramic members 30 are placed on a base 80 the upper portion of which is designed to have a V-shape in its cross-section so as to allow the porous ceramic members 30 to be stacked thereon in a tilted manner, and sealing material paste to form a sealing material layer 24 is then applied onto two side faces 30a and 30b facing upward with an even thickness to form a sealing material paste layer 81; thereafter, a laminating process for forming another porous ceramic member 30 on this sealing material paste layer 81 is successively repeated so that a rectangular columnar laminated body 30 having a predetermined size is manufactured. At this time, with respect to the porous ceramic members 30 corresponding to four corners of the laminated body of the rectangular columnar porous ceramic member 30, a triangular columnar porous ceramic member 30c, which is formed by cutting a quadrangular columnar porous ceramic member into two, is bonded to a resin member 82 having the same shape as the triangular columnar porous ceramic member 30c by using a both-sided tape with easy peelability to prepare a corner member, and these corner members are used for the four corners of the laminated body, and after the lamination processes of the porous ceramic members 30, all the resin members 82 constituting the four corners of the laminated body of the rectangular columnar ceramic member 30 are removed; thus, a laminated body of the rectangular columnar porous ceramic member 30 is allowed to have a polygonal column shape in its cross section. With this arrangement, it is possible to reduce the quantity of a waste corresponding to porous ceramic members to be disposed of, after the formation of the ceramic block by cutting the peripheral portion of the laminated body.

[0118] With respect to the method for manufacturing the laminated body having a polygonal column shape in its cross section except for the method shown in Fig. 5, for example, a method in which the porous ceramic members to be located on four corners are omitted and a method in which porous ceramic members having a triangular shape are combined with one another may be used, in accordance with the shape of a honeycomb filter to be manufactured. Here, a laminated body of a quadrangular columnar ceramic member may of course be manufactured.

[0119] Here, with respect to the material used for forming the sealing material paste, the same materials as described in the honeycomb filter of the present invention may be used; therefore, the description thereof is omitted.

[0120] Next, the laminated body of this porous ceramic member 30 is heated so that the sealing material paste layer 81 is dried and solidified to become a sealing material layer 24, and the peripheral portion of this is then cut into a shape as shown in Fig. 2 by using, for example, a diamond cutter so that a ceramic block 25 is manufactured.

[0121] Then, a sealingmaterial layer 26 is formed on the periphery of the ceramic block 25 by using the sealing material paste so that a honeycomb filter having a structure in which a plurality of porous ceramic members are combined with one another through sealing material layers is manufactured.

[0122] Each of the honeycomb filters manufactured in this manner has a column shape, and also has a structure in which a number of through holes are placed in parallel with one another with a partition wall interposed therebetween.

[0123] In the case where the honeycomb filter has a structure constituted by a single sintered body as a whole as shown in Fig. 1, the wall portion separating a number of through holes function as filters for collecting particulates as a whole; in contrast, in the case where the honeycomb filter has a structure in which a plurality of porous ceramic members are combined with one another through sealing material layers as shown in Fig. 2, since the wall portion separating a number of through holes is constituted by a partition wall forming the porous ceramic member and a sealing material layer used for combining the porous ceramic members, one portion thereof, that is, the portion of the partition wall that is not made in contact with the sealing material layer of the porous ceramic member functions as the filter for collecting particles.

burn and remove the particulates deposited on the wall portion (partition wall) in the regenerating process of the honeycomb filter 60 as described above, and with respect to the heating means 610, not particularly limited, for example, a device such as an electric heater, a burner or the like may be used.

[0139] With respect to the gas to be made to flow into the through holes, for example, exhaust gases and air are used.

[0140] Moreover, as shown in Fig. 6, the exhaust gas purifying device of this type may have a system in which the honeycomb filter 60 is heated by the heating means 610 placed on the exhaust gas inlet side of the honeycomb filter 60, or a system in which an oxidizing catalyst is supported on the honeycomb filter, with hydrocarbon being allowed to flow into the honeycomb filter supporting the oxidizing catalyst, so that the honeycomb filter is allowed to generate heat, or a system in which an oxidizing catalyst is placed on the exhaust gas inlet side of the honeycomb filter and the oxidizing catalyst is allowed to generate heat by supplying hydrocarbon to the oxidizing catalyst so that the honeycomb filter is heated.

[0141] Since the reaction between the oxidizing catalyst and hydrocarbon is a heat generating reaction, the honeycomb filter can be regenerated in parallel with the exhaust gas purifying process, by utilizing a large amount of heat generated during the reaction.

[0142] Upon manufacturing an exhaust gas purifying device in which the honeycomb filter of the present invention is installed, first, a holding sealing material with which the periphery of the honeycomb filter of the present invention is coated is prepared.

[0143] In order to form the holding sealing material, first, an inorganic mat-shaped matter (web) is formed by using inorganic fibers, such as crystalline alumina fibers, alumina-silica fibers, silica fibers and the like, and fibers and the like containing one or more kinds of these inorganic fibers.

[0144] Here, with respect to the method for forming the above-mentioned inorganic mat-shaped matter, not particularly limited, for example, a method in which the above-mentioned fibers and the like are dispersed in a solution containing an adhesive so that, by utilizing a paper machine and the like for forming paper, an inorganic mat-shaped matter is formed is proposed.

[0145] Moreover, the above-mentioned inorganic mat-shaped matter is desirably subjected to a needle punching process. This needle punching process allows the fibers to entangle with one another so that it is possible to prepare a holding sealing material that has high elasticity and is superior in the holding strength for the honeycomb filter.

[0146] Thereafter, the above-mentioned inorganic mat-shaped matter is subjected to a cutting process so that a holding sealing material, which has the above-mentioned shape in which a convex portion is formed on one side of a base portion having a rectangular shape, with a concave section being formed in the side opposing to the one side, is formed.

[0147] Next, the periphery of the honeycomb filter of the present invention is coated with the above-mentioned holding sealing material so that the holding sealing material is fixed thereon.

[0148] With respect to the means for fixing the above-mentioned holding sealing material, not particularly limited, for example, a means for bonding the holding sealing material by an adhesive or a means for tying it by using a string-shaped member may be used. Moreover, the sequence may proceed to the next process with the honeycomb filter being coated with the holding sealing material, without fixing it by using any specific means. Here, the above-mentioned string-shaped member may be made of a material to be decomposed through heat. Even if the string-shaped member is decomposed through heat after the honeycomb filter has been placed inside the casing, the holding sealing material is free from peeling as far as the honeycomb filter has already been placed inside the casing.

[0149] Next, the honeycomb filter that has been subjected to the above-mentioned processes is placed inside the casing.

[0150] Here, since the material, shape, structure and the like of the above-mentioned casing have been described earlier, the description thereof is omitted.

[0151] With respect to the method for installing the honeycomb filter in the casing, in the case where the casing is prepared as a cylindrical casing 71 (Fig. 7 (a)), for example, the following method is proposed: a honeycomb filter coated with the holding sealing material is pushed into one of its end faces, and after having been placed at a predetermined position, end faces to be connected to an introduction pipe, piping, a discharging pipe and the like are formed on the two ends of the casing 71. Here, the casing 71 may have a cylinder shape with a bottom face.

[0152] In this process, in order to prevent the secured honeycomb filter from easily moving, factors, such as the thickness of the holding sealing material, the size of the honeycomb filter, the size of the honeycomb filter and the size of the casing 71, need to be adjusted to a degree in which the pushing process can be carried out with a considerably high pressing force being applied.

[0153] Moreover, in the case where the casing is prepared as a two-division shell-shaped casing 72 as shown in Fig. 7 (b), for example, the following method is proposed: after a honeycomb filter has been placed at a predetermined position inside a lower shell 72b of half-cylinder, an upper shell 72a of half-cylinder is placed on the lower shell 72b so that through holes 73a formed in an upper fixing portion 73 and through holes 74a formed in a lower fixing portion 74 are made coincident with each other. Further, a bolt 75 is inserted through each of the through holes 73a and 74a and

Next, the ceramic dried body that had been subjected to the mouth sealing process was again dried by using a microwave drier, the resulting dried body was then degreased at 400°C, and sintered at 2200°C in a normal-pressure argon atmosphere for 4 hours to manufacture a porous ceramic member, as shown in Fig. 2, which was made of a silicon carbide sintered body, and had a size of 33 mm × 33 mm × 300 mm, the number of through holes of 31 pcs/cm² and a thickness of the partition wall of 0.3 mm.

(2) Next, a number of the porous ceramic members were combined with one another by using a heat-resistant adhesive paste containing 19.6% by weight of alumina fibers having a fiber length of 0.2 mm, 67.8% by weight of silicon carbide particles having an average particle size of 0.6 μm, 10.1% by weight of silica sol and 2.5% by weight of carboxy methyl cellulose through the method explained by reference to Fig. 5, and then cut by using a diamond cutter; thus, a cylindrical ceramic block having a diameter of 165 mm, as shown in Fig. 2, was obtained.

[0163] Next, ceramic fibers made of alumina silicate (shot content: 3%, fiber length: 0.1 to 100 mm) (23.3% by weight), which served as inorganic fibers, silicon carbide powder having an average particle size of 0.3 μm (30.2% by weight), which served as inorganic particles, silica sol (SiO₂ content in the sol: 30% by weight) (7% by weight), which served as an inorganic binder, carboxymethyl cellulose (0.5% by weight), which served as an organic binder, and water (39% by weight) were mixed and kneaded to prepare a sealing material paste.

[0164] Next, a sealing material paste layer having a thickness of 1.0 mm was formed on the peripheral portion of the ceramic block by using the above-mentioned sealing material paste. Further, this sealing material paste layer was dried at 120°C so that a cylindrical honeycomb filter, as shown in Fig. 1, was manufactured.

[0165] In the honeycomb filter thus manufactured, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 20%, and the plug had a porosity of 3%; thus, the porosity of the plug was 0.15 times as much as the porosity of the columnar body.

(Example 2)

[0166] The same processes as those of Example 1 were carried out except that a filler paste, which had been prepared by wet-mixing powder of α-type silicon carbide having an average particle size of 10 μm (60% by weight) and powder of β-type silicon carbide having an average particle size of 0.5 μm (40% by weight), and adding, to 100 parts by weight of the resulting mixture, 2 parts by weight of UNILoop, 8 parts by weight of OX-20, 1.1 parts by weight of PLYSURF, 4 parts by weight of Binder D and 0.2 parts by weight of acrylic particles so as to be evenly mixed, was used to manufacture a honeycomb filter.

[0167] In the honeycomb filter according to Example 2, thus manufactured, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 20%, and the plug had a porosity of 5%; thus, the porosity of the plug was 0.25 times as much as the porosity of the columnar body.

(Example 3)

[0168] The same processes as those of Example 1 were carried out except that a filler paste, which had been prepared by wet-mixing powder of α-type silicon carbide having an average particle size of 10 μm (60% by weight) and powder of β-type silicon carbide having an average particle size of 0.5 μm (40% by weight), and adding, to 100 parts by weight of the resulting mixture, 4 parts by weight of UNILoop, 11 parts by weight of OX-20, 2 parts by weight of PLYSURF, 5 parts by weight of Binder D and 10 parts by weight of acrylic particles so as to be evenly mixed, was used to manufacture a honeycomb filter.

[0169] In the honeycomb filter according to Example 3, thus manufactured, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 20%, and the plug had a porosity of 30%; thus, the porosity of the plug was 1.5 times as much as the porosity of the columnar body.

(Example 4)

[0170] The same processes as those of Example 1 were carried out except that a filler paste, which had been prepared by wet-mixing powder of α-type silicon carbide having an average particle size of 10 μm (70% by weight) and powder of β-type silicon carbide having an average particle size of 0.5 μm (30% by weight), and adding, to 100 parts by weight of the resulting mixture, 10 parts by weight of UNILoop, 15 parts by weight of OX-20, 3 parts by weight of PLYSURF, 8 parts by weight of Binder D and 25 parts by weight of acrylic particles so as to be evenly mixed, was used to manufacture a honeycomb filter.

[0171] In the honeycomb filter according to Example 3, thus manufactured, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 20%, and the plug had a porosity of 80%; thus, the porosity of the plug was 4.0 times as much as the porosity of the columnar body.

had an average pore diameter of 10 μm with a porosity of 50%, and the plug had a porosity of 12%; thus, the porosity of the plug was 0.24 times as much as the porosity of the columnar body.

(Example 7)

[0182] The same processes as those of Example 5 were carried out except that a filler paste, which had been prepared by wet-mixing powder of α -type silicon carbide having an average particle size of 10 μm (70% by weight) and powder of β -type silicon carbide having an average particle size of 0.5 μm (30% by weight), and adding, to 100 parts by weight of the resulting mixture, 10 parts by weight of UNILoop, 15 parts by weight of OX-20, 3 parts by weight of PLYSURF, 8 parts by weight of Binder D and 23 parts by weight of acrylic particles so as to be evenly mixed, was used to manufacture a honeycomb filter.

[0183] In the honeycomb filter according to Example 7 thus manufactured, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, and the plug had a porosity of 75%; thus, the porosity of the plug was 1.5 times as much as the porosity of the columnar body.

(Example 8)

[0184] The same processes as those of Example 5 were carried out except that a filler paste, which had been prepared by wet-mixing powder of α -type silicon carbide having an average particle size of 10 μm (70% by weight) and powder of β -type silicon carbide having an average particle size of 0.5 μm (30% by weight), and adding, to 100 parts by weight of the resulting mixture, 10 parts by weight of UNILoop, 15 parts by weight of OX-20, 3 parts by weight of PLYSURF, 8 parts by weight of Binder D and 30 parts by weight of acrylic particles so as to be evenly mixed, was used to manufacture a honeycomb filter.

[0185] In the honeycomb filter according to Example 8 thus manufactured, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, and the plug had a porosity of 90%; thus, the porosity of the plug was 1.8 times as much as the porosity of the columnar body.

(Comparative Example 3)

[0186] The same processes as those of Example 5 were carried out except that a filler paste, which had been prepared by wet-mixing powder of α -type silicon carbide having an average particle size of 10 μm (60% by weight) and powder of β -type silicon carbide having an average particle size of 0.5 μm (40% by weight), and adding, to 100 parts by weight of the resulting mixture, 2 parts by weight of UNILoop, 8 parts by weight of OX-20, 1.1 parts by weight of PLYSURF, 4 parts by weight of Binder D and 0.2 parts by weight of acrylic particles so as to be evenly mixed, was used to manufacture a honeycomb filter.

[0187] In the honeycomb filter according to Comparative Example 3 thus manufactured, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, and the plug had a porosity of 5%; thus, the porosity of the plug was 0.1 times as much as the porosity of the columnar body.

(Comparative Example 4)

[0188] The same processes as those of Example 5 were carried out except that a filler paste, which had been prepared by wet-mixing powder of α -type silicon carbide having an average particle size of 10 μm (70% by weight) and powder of β -type silicon carbide having an average particle size of 0.5 μm (30% by weight), and adding, to 100 parts by weight of the resulting mixture, 10 parts by weight of UNILoop, 15 parts by weight of OX-20, 3 parts by weight of PLYSURF, 8.3 parts by weight of Binder D and 33 parts by weight of acrylic particles so as to be evenly mixed, was used to manufacture a honeycomb filter.

[0189] In the honeycomb filter according to Comparative Example 4 thus manufactured, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, and the plug had a porosity of 92%; thus, the porosity of the plug was 1.84 times as much as the porosity of the columnar body.

(Example 9)

[0190] The same processes as those of Example 1 were carried out except that the material paste and the filler paste were prepared in the following manner to manufacture a honeycomb filter.

(Comparative Example 6)

[0200] The same processes as those of Example 9 were carried out except that a filler paste, which had been prepared by wet-mixing powder of α -type silicon carbide having an average particle size of 10 μm (70% by weight) and powder of β -type silicon carbide having an average particle size of 0.5 μm (30% by weight), and adding, to 100 parts by weight of the resulting mixture, 10 parts by weight of UNILoop, 15 parts by weight of OX-20, 3 parts by weight of PLYSURF, 8.3 parts by weight of Binder D and 33 parts by weight of acrylic particles so as to be evenly mixed, was used to manufacture a honeycomb filter.

[0201] In the honeycomb filter according to Comparative Example 6 thus manufactured, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 80%, and the plug had a porosity of 92%; thus, the porosity of the plug was 1.15 times as much as the porosity of the columnar body.

[0202] With respect to the honeycomb filters according to Examples 1 to 11 and Comparative Examples 1 to 6 manufactured as described above, the porosity (%) of the columnar body, the porosity (%) of the plug and the multiplying ratio of the porosity of the plug to the porosity of the columnar body are collectively shown in Table 1.

[0203] Moreover, with respect to the honeycomb filters according to Examples 1 to 11 and Comparative Examples 1 to 6 that had been sintered, it was confirmed whether or not there was any gap between the plug and the partition wall as well as whether or not there was any crack occurring in the plug and a portion of the partition wall contacting the plug, and the honeycomb filters in which neither a gap nor a crack occurred were subjected to an endurance test in which each of the honeycomb filters according to the respective examples and comparative examples was placed in an exhaust gas purifying device as shown in Fig. 6, which was installed in an exhaust passage of an engine, and the engine was driven at the number of revolutions of 3000 min^{-1} with a torque of 50 Nm for 10 hours so that an exhaust gas purifying process was carried out. After the above-mentioned endurance test, each of the honeycomb filters was taken out and visually observed as to whether or not any cracks occurred. Moreover, the honeycomb filters that had no cracks after the endurance test were further subjected to heat cycling tests in which the above-mentioned endurance tests were repeated 300 times, and each of the honeycomb filters was taken out and visually observed as to whether or not any cracks occurred.

[0204] The results are shown in the following Table 1.

[0205] As shown in Table 1, in any one of the honeycomb filters according to Examples 2, 3, 6, 7, 10 and 11, no gap was observed between the plug and the partition wall, and no cracks were observed in the plug and a portion of the partition wall contacting the plug, in any of the cases after the firing process, after the endurance test and after heat cycling tests. Moreover, in any one of the honeycomb filters according to Examples 1, 4, 5, 8 and 9, no gap was observed between the plug and the partition wall, and no cracks were observed in the plug and a portion of the partition wall contacting the plug, after the firing process as well as after the endurance test; however, after the heat cycling tests, cracks were observed.

[0206] In contrast, in the honeycomb filters according to Comparative Examples 1, 3 and 5, no cracks or the like were observed between the plug and the partition wall after the firing process; however cracks were observed in the plug and a portion of the partition wall contacting the plug after the endurance test. Moreover, in the honeycomb filter according to Comparative Example 2, a gap was observed between the plug and the partition wall after the firing process, and in the honeycomb filters according to Comparative Examples 4 and 6, cracks were observed in the plug after the firing process.

[0207] The results of the evaluation tests of the honeycomb filters according to Examples 1 to 11 show that those honeycomb filters in which the columnar body has a porosity in a range from 20 to 80% and the plug has a porosity of 90% or less, with the porosity being 0.15 to 4.0 times as much as the porosity of the columnar body are free from a gap occurring between the plug and the partition wall as well as occurrence of cracks in the plug and a portion of the partition wall contacting the plug, after the firing process and at the time of heating; however, the results of the evaluation tests of the honeycomb filters according to Examples 1, 4, 5 and 8 as well as 9 show that those filters in which the porosity of the plug is less than 0.25 times as much as the porosity of the columnar body, or exceeds 1.5 times as much as the porosity of the columnar body tend to cause cracks in the plug and a portion of the partition wall contacting the plug, after a long-term repetitive use.

[0208] Further, the results of the evaluation tests of the honeycomb filters according to Comparative Examples 1, 3 and 5 show that in the case where the porosity of the plug is less than 0.15 times as much as the porosity of the columnar body, even if no cracks occur in the plug and a portion in the partition wall contacting the plug, the difference between the coefficient of thermal expansion of the columnar body and the coefficient of thermal expansion of the plug becomes greater, with the result that, when the columnar body and the plug are heated by high-temperature exhaust gases, thermal stresses are accumulated between the two members, resulting in cracks in the plug and a portion of the partition wall contacting the plug.

[0209] Moreover, the results of the evaluation tests of the honeycomb filter according to Comparative Example 2 show that when the porosity of the plug exceeds 4.0 times as much as the porosity of the columnar body, a gap tends to occur between the plug and a portion of the partition wall contacting the plug after the firing process.

[0210] Furthermore, the results of the evaluation tests of the honeycomb filters according to Comparative Examples 4 and 6 show that in the case where the porosity of the plug exceeds 90%, even if the porosity of the plug is 0.15 to 4.0 times as much as the porosity of the columnar body, the strength of the plug is lowered to cause cracks in the plug and a portion in the partition wall contacting the plug during the firing process.

[0211] In this manner, any of the honeycomb filters according to Comparative Examples 1 to 6 fail to sufficiently function as a filter.

(Example 12)

[0212]

(1) The same processes as Example 1 were carried out to manufacture a honeycomb filter, in which: the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 20%, the plug had a porosity of 3%, and the porosity of the plug was 0.15 times as much as the porosity of the columnar body.

(2) Powder of $\gamma\text{-Al}_2\text{O}_3$ ground to a particle size of 5 μm or less was put into 1,3-butane diol, and this was stirred at 60°C for 5 hours so that a 1,3-butane diol solution in a slurry state, which contained 3% by weight of alumina, was prepared. The honeycomb filter was immersed in this 1,3-butane diol solution, and this was then heated at 150°C for 2 hours, at 400°C for 2 hours and at 700°C for 8 hours so that an alumina layer serving as a catalyst supporting film was formed on the surface of the honeycomb filter (columnar body except for a plug, and the plug) at a rate of 1 g/L.

[0213] Diammine dinitro platinum nitric acid ($[\text{Pt}(\text{NH}_3)_2(\text{NO}_2)_2]\text{HNO}_3$) having a platinum concentration of 4.53% by weight was diluted in distilled water, and the honeycomb filter having a water absorbing amount of 28.0 g/L was immersed therein so that Pt was deposited thereon at a rate of 2 g/L, and this was then heated at 110°C for 2 hours, and heated at 500°C for one hour in a nitrogen atmosphere so that a honeycomb filter having a platinum catalyst deposited on the surface thereof was manufactured.

on the surface thereof was manufactured.

[0223] In the honeycomb filter having the platinum catalyst deposited on the surface thereof, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 20%, the plug had a porosity of 24%, and the porosity of the plug was 1.20 times as much as the porosity of the columnar body.

(Example 16)

[0224]

(1) The same processes as Example 3 were carried out to manufacture a honeycomb filter, in which: the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 20%, the plug had a porosity of 30%, and the porosity of the plug was 1.50 times as much as the porosity of the columnar body.

(2) The same processes as Example 12 were carried out so that an alumina layer serving as a catalyst supporting film was formed on the surface of a honeycomb filter (columnar body except for a plug, and the plug) at a rate of 1 g/L; thus, a honeycomb filter having a platinum catalyst deposited on the surface thereof was manufactured.

[0225] In the honeycomb filter having the platinum catalyst deposited on the surface thereof, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 20%, the plug had a porosity of 30%, and the porosity of the plug was 1.50 times as much as the porosity of the columnar body.

(Example 17)

[0226]

(1) The same processes as Example 4 were carried out to manufacture a honeycomb filter, in which: the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 20%, the plug had a porosity of 80%, and the porosity of the plug was 4.00 times as much as the porosity of the columnar body.

(2) The same processes as Example 12 were carried out so that an alumina layer serving as a catalyst supporting film was formed on the surface of a honeycomb filter (columnar body except for a plug, and the plug) at a rate of 1 g/L; thus, a honeycomb filter having a platinum catalyst deposited on the surface thereof was manufactured.

[0227] In the honeycomb filter having the platinum catalyst deposited on the surface thereof, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 20%, the plug had a porosity of 80%, and the porosity of the plug was 4.00 times as much as the porosity of the columnar body.

(Example 18)

[0228]

(1) The same processes as Example 5 were carried out to manufacture a honeycomb filter, in which: the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, the plug had a porosity of 7.5%, and the porosity of the plug was 0.15 times as much as the porosity of the columnar body.

(2) The same processes as Example 12 were carried out so that an alumina layer serving as a catalyst supporting film was formed on the surface of a honeycomb filter (columnar body except for a plug, and the plug) at a rate of 1 g/L; thus, a honeycomb filter having a platinum catalyst deposited on the surface thereof was manufactured.

[0229] In the honeycomb filter having the platinum catalyst deposited on the surface thereof, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, the plug had a porosity of 7.5%, and the porosity of the plug was 0.15 times as much as the porosity of the columnar body.

(Example 19)

[0230]

(1) The same processes as Example 6 were carried out to manufacture a honeycomb filter, in which: the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, the plug had a porosity of 12%, and the porosity of the plug was 0.24 times as much as the porosity of the columnar body.

(2) The same processes as Example 12 were carried out so that an alumina layer serving as a catalyst supporting

body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, the plug had a porosity of 75%, and the porosity of the plug was 1.50 times as much as the porosity of the columnar body.

(2) The same processes as Example 12 were carried out so that an alumina layer serving as a catalyst supporting film was formed on the surface of a honeycomb filter (columnar body except for a plug, and the plug) at a rate of 1 g/L; thus, a honeycomb filter having a platinum catalyst deposited on the surface thereof was manufactured.

[0240] In the honeycomb filter having the platinum catalyst deposited on the surface thereof, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, the plug had a porosity of 75%, and the porosity of the plug was 1.50 times as much as the porosity of the columnar body.

(Example 23)

[0241]

(1) The same processes as Example 8 were carried out to manufacture a honeycomb filter, in which: the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, the plug had a porosity of 90%, and the porosity of the plug was 1.80 times as much as the porosity of the columnar body.

(2) The same processes as Example 12 were carried out so that an alumina layer serving as a catalyst supporting film was formed on the surface of a honeycomb filter (columnar body except for a plug, and the plug) at a rate of 1 g/L; thus, a honeycomb filter having a platinum catalyst deposited on the surface thereof was manufactured.

[0242] In the honeycomb filter having the platinum catalyst deposited on the surface thereof, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 50%, the plug had a porosity of 90%, and the porosity of the plug was 1.80 times as much as the porosity of the columnar body.

(Example 24)

[0243]

(1) The same processes as Example 9 were carried out to manufacture a honeycomb filter, in which: the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 80%, the plug had a porosity of 12%, and the porosity of the plug was 0.15 times as much as the porosity of the columnar body.

(2) The same processes as Example 12 were carried out so that an alumina layer serving as a catalyst supporting film was formed on the surface of a honeycomb filter (columnar body except for a plug, and the plug) at a rate of 1 g/L; thus, a honeycomb filter having a platinum catalyst deposited on the surface thereof was manufactured.

[0244] In the honeycomb filter having the platinum catalyst deposited on the surface thereof, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 80%, the plug had a porosity of 12%, and the porosity of the plug was 0.15 times as much as the porosity of the columnar body.

(Example 25)

[0245]

(1) The same processes as Example 10 were carried out to manufacture a honeycomb filter, in which: the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 80%, the plug had a porosity of 20%, and the porosity of the plug was 0.25 times as much as the porosity of the columnar body.

(2) The same processes as Example 12 were carried out so that an alumina layer serving as a catalyst supporting film was formed on the surface of a honeycomb filter (columnar body except for a plug, and the plug) at a rate of 1 g/L; thus, a honeycomb filter having a platinum catalyst deposited on the surface thereof was manufactured.

[0246] In the honeycomb filter having the platinum catalyst deposited on the surface thereof, the columnar body except for the plug had an average pore diameter of 10 μm with a porosity of 80%, the plug had a porosity of 20%, and the porosity of the plug was 0.25 times as much as the porosity of the columnar body.

(Example 26)

[0247] The same processes as those of Example 9 were carried out except that a filler paste, which had been pre-

except for the plug had an average pore diameter of 10 μm with a porosity of 80%, the plug had a porosity of 90%, and the porosity of the plug was 1.13 times as much as the porosity of the columnar body.

[0257] With respect to the honeycomb filters according to Examples 12 to 28 manufactured as described above, the porosity (%) of each of the columnar body and the plug prior to the alumina application, the multiplying ratio of a porosity of the plug to the porosity of the columnar body prior to the alumina application, the amount of alumina application (g/L) of each of the columnar body and the plug, the porosity (%) of each of the columnar body and the plug after the alumina application and the multiplying ratio of a porosity of the plug to the porosity of the columnar body after the alumina application are collectively shown in the following Table 2.

[0258] Moreover, with respect to the honeycomb filters according to Examples 12 to 28 that had been fired, it was confirmed whether or not there was any gap between the plug and the partition wall as well as whether or not there was any crack occurring in the plug and a portion of the partition wall contacting the plug.

[0259] With respect to the honeycomb filters in which neither a gap nor a crack occurred were subjected to an endurance test in which each of the honeycomb filters was placed in an exhaust gas purifying device as shown in Fig. 6, which was installed in an exhaust passage of an engine, and the engine was driven at the number of revolutions of 3000 min^{-1} with a torque of 50 Nm for 10 hours so that an exhaust gas purifying process was carried out. After the above-mentioned endurance test, each of the honeycomb filters was taken out and visually observed as to whether or not any cracks occurred.

[0260] Moreover, the honeycomb filters that had no cracks after the endurance test were further subjected to heat cycling tests in which the above-mentioned endurance tests were repeatedly carried out, and after having been repeatedly subjected to the endurance tests of 100 times, the resulting filters were further subjected to the endurance tests of 300 times, and each of the honeycomb filters was then taken out and visually observed as to whether or not any cracks occurred.

[0261] The results are shown in the following Table 2. The results of Comparative Examples 1 to 6 are also shown in Table 2 by reference.

[0262] As shown in Table 2, in any one of the honeycomb filters according to Examples 14, 15, 20 and 21 as well as 26 to 28, no gap was observed between the plug and the partition wall, or no cracks were observed in the plug and a portion of the partition wall contacting the plug, in any of the cases after the firing process, after the endurance test and after heat cycling tests. Moreover, in any one of the honeycomb filters according to Examples 13, 16, 19, 22 and 25, no gap was observed between the plug and the partition wall, or no cracks were observed in the plug and a portion of the partition wall contacting the plug, in any of the cases after the firing process, after the endurance test, and after the repeated endurance tests of 100 times in the heat cycling tests; however, after the repeated endurance tests of 300 times in the heat cycling tests, cracks were observed. Furthermore, in any one of the honeycomb filters according to Examples 12, 17, 18, 23 and 24, no gap was observed between the plug and the partition wall, or no cracks were observed in the plug and a portion of the partition wall contacting the plug, after the firing process as well as after the endurance tests; however, after the repeated endurance tests of 100 times in the heat cycling tests, cracks were observed.

[0263] The results of the evaluation tests of the honeycomb filters according to Examples 12 to 28 show that those honeycomb filters in which the columnar body has a porosity in a range from 20 to 80% and the plug has a porosity of 90% or less, with the porosity being 0.15 to 4.0 times as much as the porosity of the columnar body, are free from a gap occurring between the plug and the partition wall as well as occurrence of cracks in the plug and a portion of the partition wall contacting the plug, after the firing process and at the time of heating, even when an alumina layer serving as a catalyst supporting film is formed thereon; however, the results of the evaluation tests of the honeycomb filters according to Examples 12, 13, 16 to 19 as well as 22 to 25 show that those filters in which the porosity of the plug is less than 0.25 times as much as the porosity of the columnar body, or exceeds 1.5 times as much as the porosity of the columnar body tend to cause cracks in the plug and a portion of the partition wall contacting the plug, after a long-term repetitive use.

INDUSTRIAL APPLICABILITY

[0264] The honeycomb filter for purifying exhaust gases in accordance with the present invention, which has the above-mentioned arrangement, is free from a gap occurring between the plug and the partition wall and cracks occurring in the plug and a portion of the partition wall contacting the plug, during the manufacturing process as well as during use; thus, it becomes possible to provide a filter that is superior in durability.

Claims

1. A honeycomb filter for purifying exhaust gases which has a structure in which:

a columnar body made of porous ceramic comprises a number of through holes, said through holes being placed in parallel with one another in the length direction with a wall portion interposed therebetween; predetermined through holes of said through holes are filled with plugs at one end of said columnar body, while the through holes that have not been filled with said plugs at said one end are filled with plugs at the other end of said columnar body; and
a part or all of said wall portion functions as a filter for collecting particulates

wherein

the porosity of said columnar body is in a range from 20 to 80%, and the porosity of said plug is 90% or less and is also set to 0.15 to 4.0 times as much as the porosity of said columnar body.

2. The honeycomb filter for purifying exhaust gases according to claim 1,
wherein the porosity of the plug is set to 0.25 to 1.5 times as much as the porosity of the columnar body.
3. The honeycomb filter for purifying exhaust gases according to claim 1 or 2,
wherein a catalyst is supported thereon.
4. The honeycomb filter for purifying exhaust gases according to any one of claims 1 to 3,
wherein a catalyst supporting film is placed on the surface thereof.
5. The honeycomb filter for purifying exhaust gases according to any one of claims 1 to 4,
wherein, as a method for removing fine particles that have been collected and accumulated, a back-washing process using a gas flow is adopted.

Fig. 1

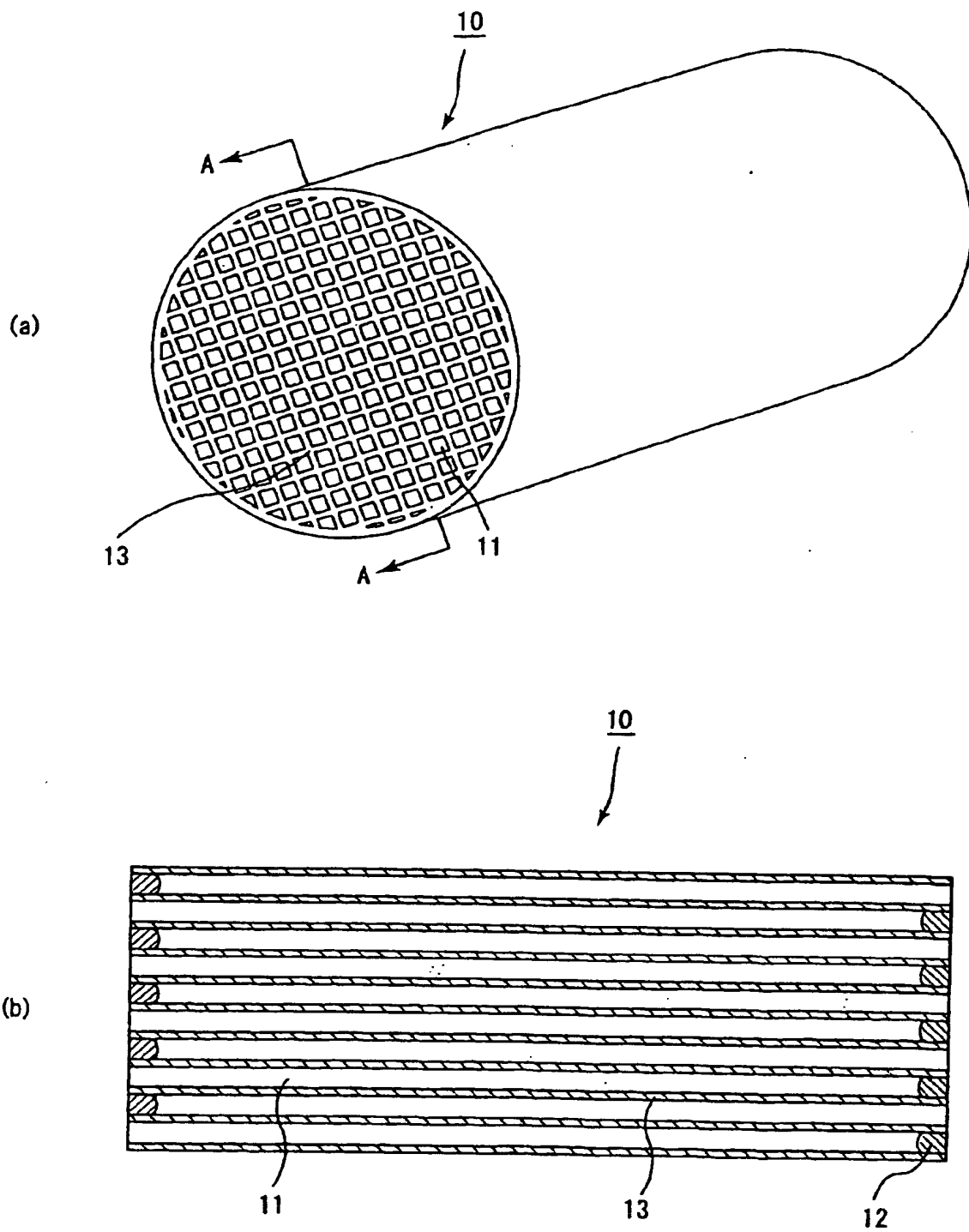


Fig. 3

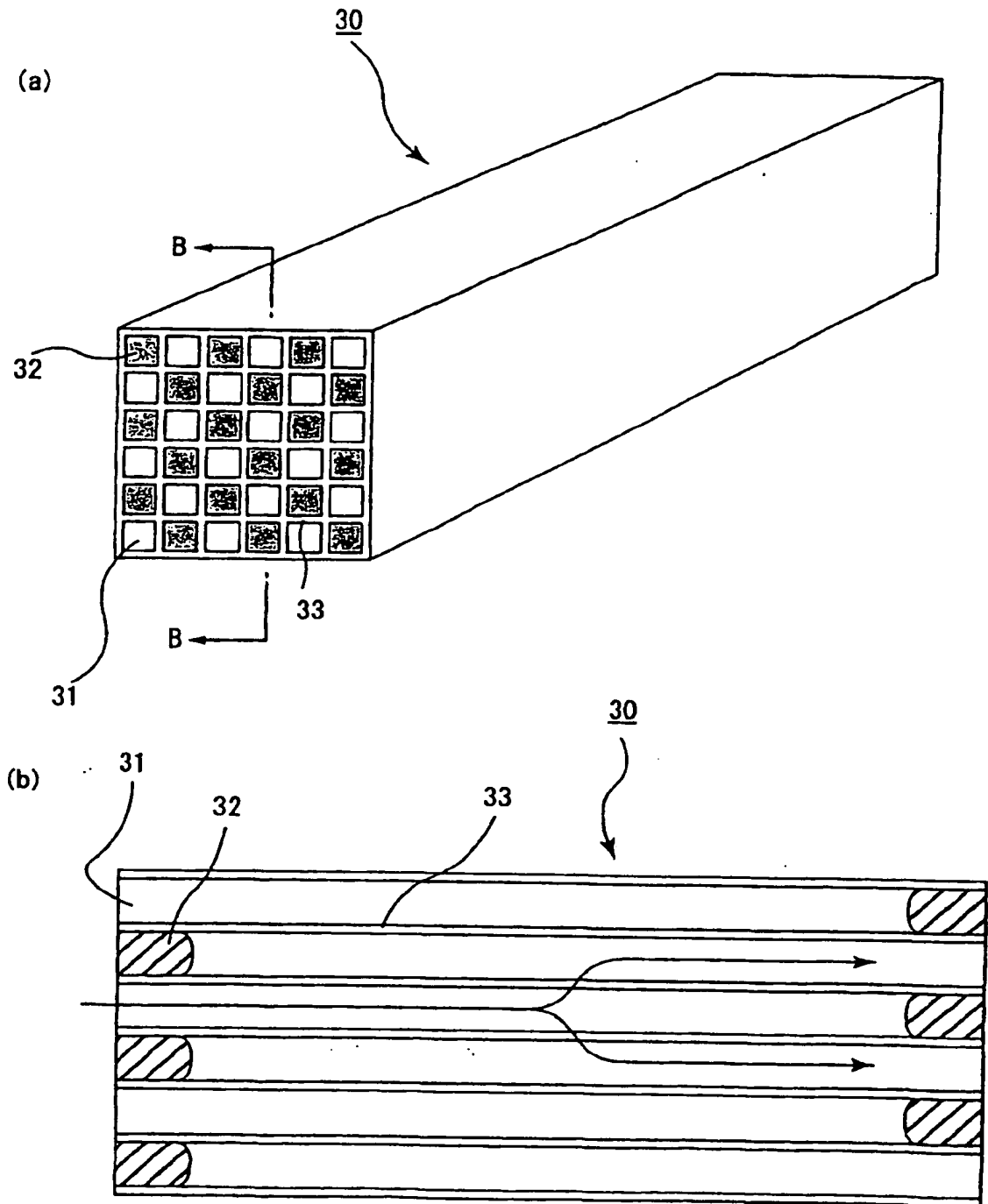


Fig. 5

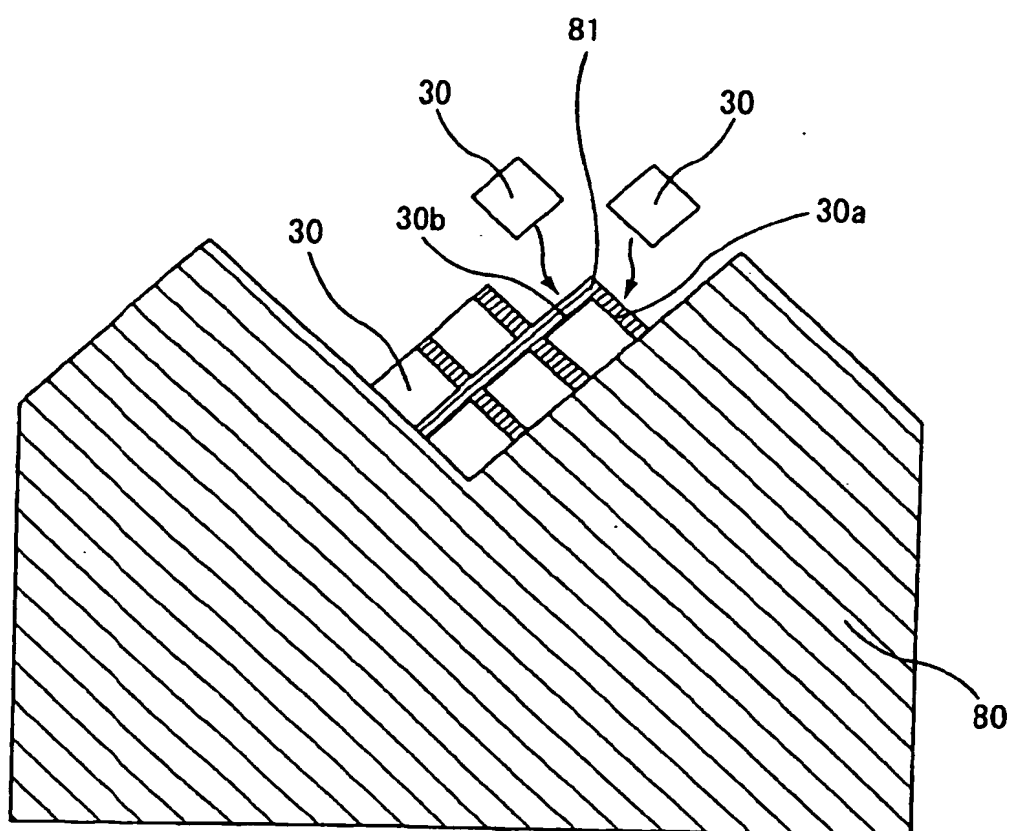
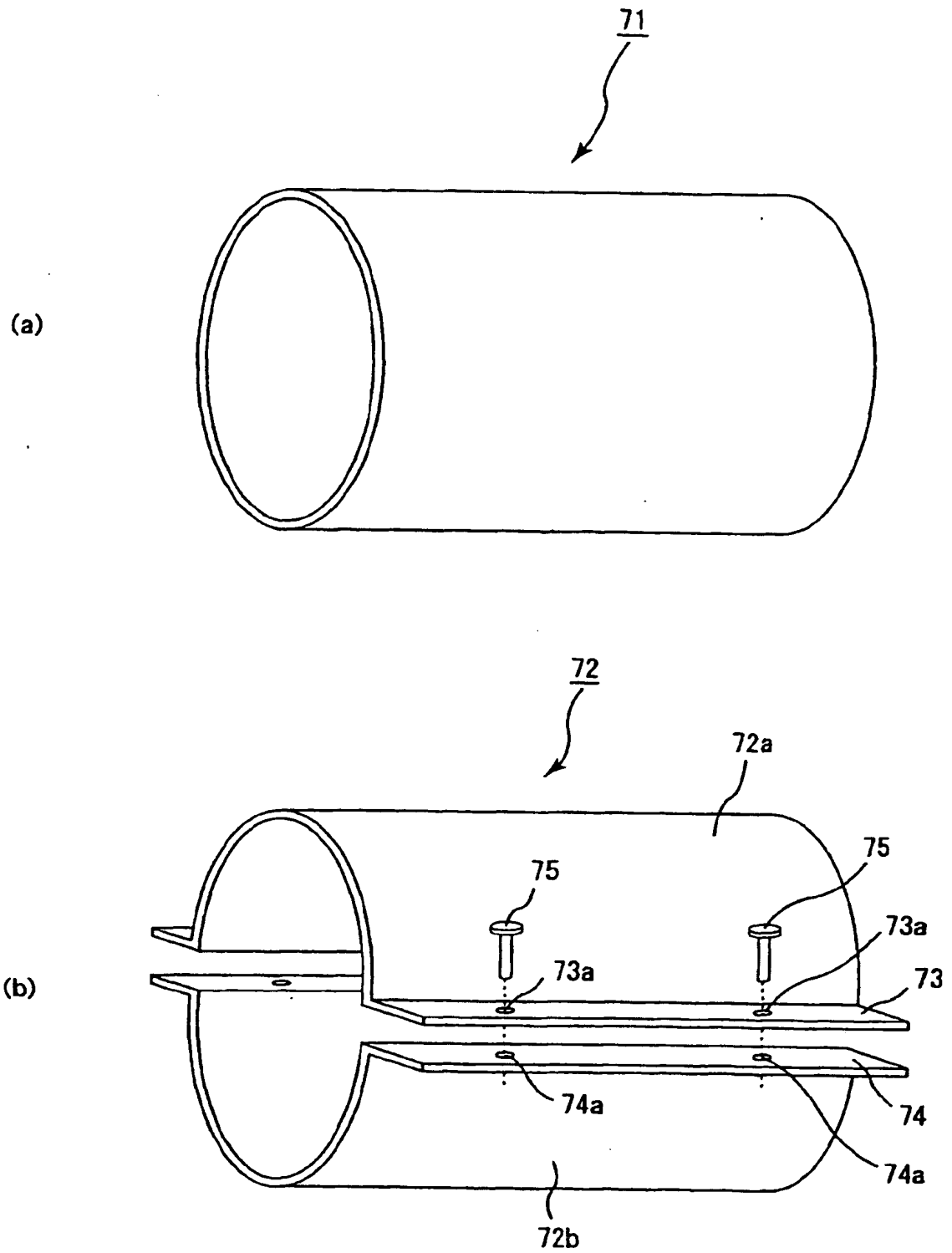


Fig. 7



INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP03/04480

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	Microfilm of the specification and drawings annexed to the request of Japanese Utility Model Application No. 146426/1984 (Laid-open No. 62217/1986) (Toyota Motor Corp.), 26 April, 1986 (26.04.86), Full text (Family: none)	1-6

Form PCT/ISA/210 (continuation of second sheet) (July 1998)